Journal of Physics: Conference Series

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To cite this article: A I Menshakov and P A Skorynina 2019 *J. Phys.: Conf. Ser.* **1393** 012109

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Journal of Physics: Conference Series **1393** (2019) 012109 doi:10.1088/1742-6596/1393/1/012109

A new method of low-temperature cementation of stainless steel by decomposition of C_2H_2 in low-energy electron beam generated plasma

A I Menshakov[1,2] **and P A Skorynina**[3]

[1] Institute of Electrophysics UD RAS, 106 Amundsen Str., Yekaterinburg, 620016, Russia
[2] Ural Federal University, 19 Mira Str., Yekaterinburg, 620002, Russia
[3] Institute of Engineering Science UD RAS, 34 Komsomolskaya Str., Yekaterinburg, 620049, Russia

E-mail: aim@iep.uran.ru

Abstract. The method of low-temperature (400–500°C) cementation of AISI430 stainless steel by decomposition of acetylene in a wide (100 cm^2) low-energy (200–300 eV) electron beam generated plasma in an $Ar+C_2H_2$ gas mixture was investigated. The composition of a beam $Ar+C_2H_2$-plasma is investigated and it is shown that the degree of decomposition of acetylene varies with the current and energy of the electron beam. It is shown that the magnitude of the flow of acetylene significantly affects the formation rate and hardness of the hardened layer. From the obtained results it can be seen that at fixed values of argon pressure (~ 0.8 mTorr), beam current (3.5 A), bias voltage (–120 V), sample temperature (500°C) and exposure time (3 h) increase in Q_{C2H2} from 1 to 4–5 $cm^3 \cdot min^{-1}$ leads to an increase in the thickness and microhardness of the hardened layer. With a further increase in Q_{C2H2}, an abrupt decrease in the rate of formation of the hard layer occurs.

1. Introduction

Plasma cementation is one of the types of chemical heat treatment, and consists in the diffusion saturation of the surface layer of the products with the alloying element – carbon. This allows you to form a modified layer near the surface, resulting in significantly improved product characteristics such as hardness, wear resistance, resistance to stamina, fatigue strength, corrosion resistance.

Usually for plasma cementation, facilities based on a glow discharge are used [1], and methane (CH_4), acetylene (C_2H_2), and propane (C_3H_8) are most commonly used as a saturating gaseous medium [2–4]. An alternative method of generating plasma for different materials processing methods is the use of electron beams.

The advantage of using a low-energy electron beam for plasma generation is the ability to set the electron energy corresponding to the maximum ionization cross section of the working gas atoms by electron impact, which will increase the efficiency of plasma generation compared to traditional gas-discharge systems. The authors of [5] developed a method of nitriding in the plasma of a low-energy electron beam, which not only generated the plasma, but also heated the workpiece. A plasma emitter of electrons with grid stabilization based on a glow discharge with a hollow cathode was used as an electron source, and the cross-sectional area of the beam was ~ 80 cm^2. An important advantage of such a source for technological applications is the independent control of the emission current and energy of electrons, the pressure and composition of the gaseous medium in the working chamber over

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a wide range, which provides flexible control over the parameters of the generated plasma. The main advantage of heating by an electron beam in comparison with the treatment in a glow discharge is the absence of sputtering of the surface by a high-energy ion flux, which makes it possible to ensure the required level of surface cleanliness and to increase the efficiency of processing products.

However, this approach was not previously used for cementation, and the promise of this method as applied to cementation is due to the fact that electrons accelerated to energy of the order of 100–200 eV more efficiently than gas discharge provide excitation, ionization and dissociation of molecules and atoms of the gaseous medium.

This work is devoted to the study of a new method of surface cementation of steels and alloys by decomposing acetylene in the plasma of a low-energy electron beam and heating products to the required temperature with a wide electron beam.

2. Experimental technique

The samples were processed in an argon-acetylene plasma generated by a low-energy electron beam, using a two-step source of a wide (D = 100 mm) electron beam with a mesh plasma cathode. Samples of size 20×10×1 mm^3 were made of AISI430 sheet steel, the steel was in rolled state and in the structure of 100 % alpha phase. Before being placed in the vacuum chamber, the samples were cleaned in an ultrasonic solvent bath. The experiment is shown in figure 1. At the initial stage, a glow discharge was ignited in argon (30 cm^3·min^{-1}), after which an accelerating voltage was applied between the grid and the discharge chamber (U_2). A bias voltage was applied to the sample table (-350 V relative to the discharge chamber) and ion cleaning and heating of the samples was carried out for 30 minutes. At the next stage, acetylene was injected into the chamber (Q_{C2H2} = 1–10 cm^3·min^{-1}) and beam parameters were set (current I_2, voltage U_2) providing heating to the required temperature (400 or 500°C), after which the samples were set at steady state mode for 3 hours. The total pressure of the gas mixture was ~ 1·10^{-3} Torr.

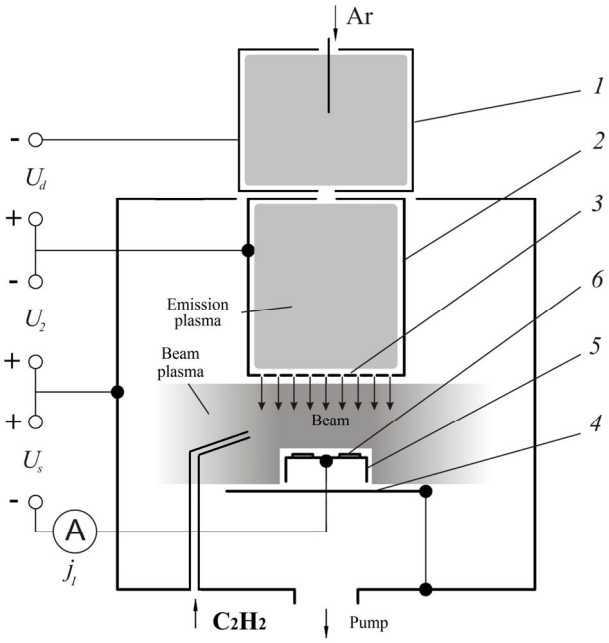

Figure 1. The scheme of the experimental setup. *1* – hollow cathode, *2* – hollow anode, *3* – grid, *4* – beam collector, *5* – insulated table, *6* – samples.

Plasma analysis by the method of optical emission spectroscopy (OES) was carried out using HR-2000 (OceanOptics) spectrograph with operating spectral range 200–1100 nm and optical resolution ~ 0,8 nm. Microhardness of the obtained samples was measured by Shimadzu DUH-211s dynamic microhardnesser with load of 10 mN. Averaging of microhardness values was carried out for 6–8 measurements. Thickness of the hardened surface layers was measured by microhardness distribution in cross section of samples. Diffraction patterns were taken on a D8 DISCOVER

diffractometer on copper radiation (Cu Kα1,2 λ = 1,542Å) with a graphite monochromator on a diffracted beam, their processing was performed using the TOPAS 3 full-profile analysis program.

3. Experimental results and discussion

The composition of the electron beam plasma in an Ar+C_2H_2 gas mixture was analyzed using optical emission spectroscopy. In the spectrum of a beam plasma, intense lines of an argon atom are emitted in the red and near-IR ranges (figure 2). In the visible part of the spectrum, there are lines of argon ions, lines of the hydrogen atom, characteristic of the Balmer series (H*, 656.2 nm), as well as molecular bands of C_2 and CH (figure 2 shows the characteristic spectrum of the transition $d^3\Pi$–$a^3\Pi^u$ (Swan system) of the C_2 molecule). The inset shows the transition band $A^2\Delta$–$X^2\Pi$ of the CH molecule. The presence of H*, C_2 and CH lines in the plasma spectrum indicates an intensive decomposition of C_2H_2 molecules in a beam plasma, and the intensity of these lines, and accordingly the decomposition intensity, increases with both an increase in beam current and an increase in electron beam energy (figure 3).

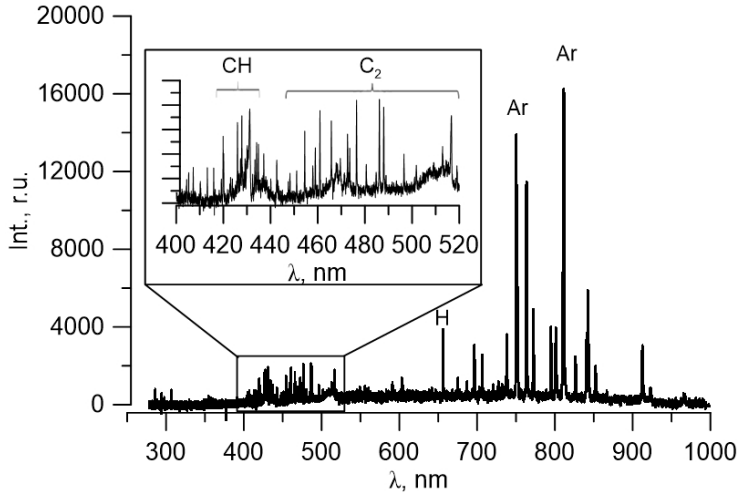

Figure 2. The spectra of electron beam plasma in Ar+C_2H_2 gas mixture. The beam current 4 A, beam energy 200 eV, Q_{C2H2} = 5 sccm.

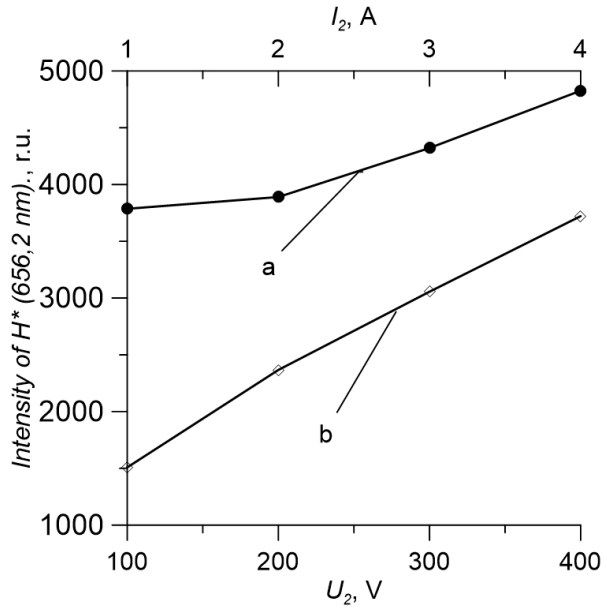

Figure 3. Dependences of I_H intensities on electron beam energy (a), (I_2 = 4 A) and beam current (b), (U_2 = 200 V). Q_{C2H2} = 10 $cm^3 \cdot min^{-1}$.

In [6], the composition of the acetylene-argon plasma of a low-pressure arc discharge was investigated. As a result, typical plasma-chemical reactions occurring in this medium were obtained, and on the basis of experimental data a corresponding model was constructed and the frequency

distribution of decomposition of acetylene molecules was obtained. According to the calculations obtained, a key role in the decomposition of C_2H_2 molecules is played by argon ions, and the reaction itself occurs in steps. At the first stage, recombination occurs with the participation of argon ions and acetylene molecules, and then decomposition of ionized and excited acetylene molecules with participation of plasma electrons takes place:

$$Ar^+ + C_2H_2 \rightarrow Ar + C_2H_2^{+,*}$$

$C_2H_2^{+,*} + e \rightarrow C_2H + H$	26%
$\rightarrow C_2 + H + H$	41%
$\rightarrow 2CH$	7%
$\rightarrow CH_2 + C$	26%
$\rightarrow C_2 + H_2$	0%

That is, according to the model, the key role in the decomposition of acetylene is played by argon ions, and since the electron beam energy is set near the maximum of the ionization cross section of argon, the decomposition intensity and acetylene use efficiency in the plasma of a low-energy electron beam will be higher than in a gas-discharge plasma.

In the technology of ion-plasma nitriding, the presence of hydrogen in the gaseous medium, which is necessary to bind residual oxygen, which forms dynamic oxide films that block the flow of active nitrogen to the surface of the part, eliminated ion etching and reduced the energy of the ion flux to the samples during nitriding [7] to a few volts, which provided a lower level of surface roughness. The advantage of using acetylene for cementation is the presence of hydrogen atoms in the decomposition products of the hydrogen that binds residual oxygen, but the decomposition of acetylene produces carbon-containing particles deposited on all surfaces of the electrode system. This necessitated the ion etching of the surface even in the presence of hydrogen in a gaseous medium. The bias potential on the samples during the processing was -(120–140) V relative to the beam collector for etching the barrier layer that blocks the entry of active particles to the treated surface, because at lower bias voltages the forming carbon-containing surface layer blocked their entry to the surface being processed, which is significant reduced the characteristics of the hardened layer.

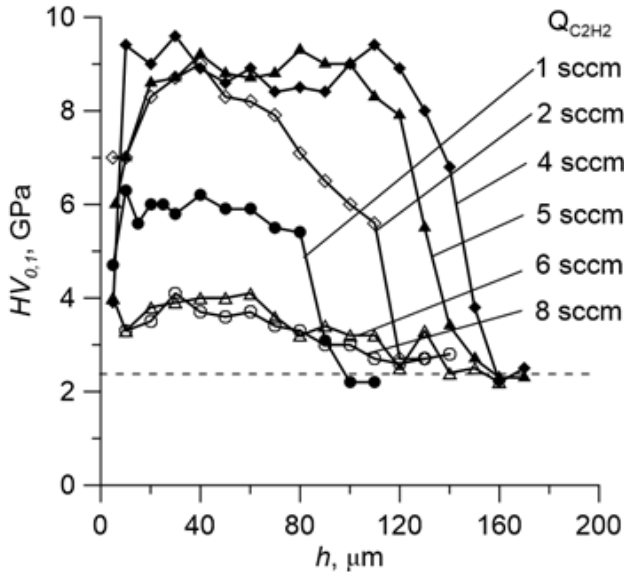

Figure 4. The microhardness profiles of hardened samples at different acetylene flow rates. P_{Ar} = 0.8 mTorr, beam current 3.5 A, bias voltage -120 V, sample temperature 500°C, exposure time 3 h.

In the course of the work, it was found that the flow rate of acetylene is significantly affected by the formation rate and hardness of the hardened layer. Figure 4 shows the microhardness profiles of hardened specimens obtained for different values of acetylene flux. From the obtained results it can be seen that with fixed values of argon pressure (~ 0.8 mTorr), beam current (3.5 A), bias voltage (–120 V), sample temperature (500°C) and exposure time (3 h) acetylene from 1 to 4–5 $cm^3 \cdot min^{-1}$ leads to an increase in the thickness and microhardness of the hardened layer to 160 μm and 9.5 GPa,

respectively. With a further increase in Q_{C2H2}, an abrupt decrease in the rate of formation of the solid layer occurs. One explanation for the nature of the dependence of the properties of the hardened layer on the acetylene flux may be that at low Q_{C2H2} fluxes, an increase in the layer growth rate is achieved due to an increase in the concentration gradient of active particles on the sample surface, while the flow rate of active particles to the surface does not exceed the entrainment rate particles by ion etching the surface and diffusing carbon atoms into the sample volume. When more active carbon particles enter the surface than are carried away by ion etching and diffusion into the reinforced volume, a carbon film is formed on the surface, which separates the active saturating medium from the material to be hardened, and the layer does not form.

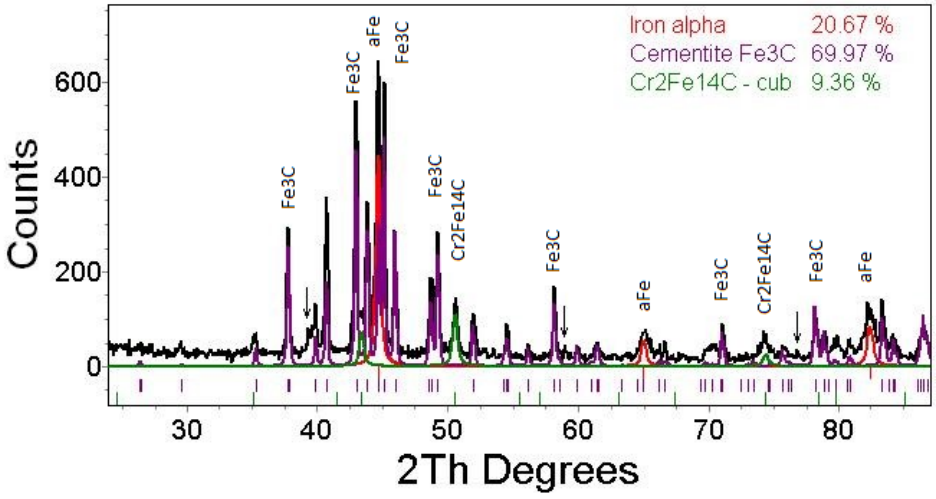

Figure 5. Difraction pattern of carbuzed surface. T = 500°C, Q_{C2H2} = 4 cm^3·min^{-1}.

According to X-ray analysis (figure 5) of the surface of the processed samples, crystalline phases α-Fe (cubic, period a = 2.8664 Å), $Cr_2Fe_{14}C$ (cubic, period a = 7.2000 Å), Fe_3C (orthorhombic, periods a = 5,091 Å, b = 6,7434 Å, c = 4,526 Å), which are solid solutions based on these structures. Moreover, the relative content of these phases significantly depends on the concentration of acetylene in the plasma (table 1). So the maximum content of Fe_3C in the hardened layer was found at an acetylene flow of 4 cm^3·min^{-1}, which corresponds to the maximum values of the thickness and hardness of the hardened layer.

Table 1. Phase composition of hardened layers.

T (°C)	Q_{C2H2} (sccm)	α-Fe (wt. %)	$Cr_2Fe_{14}C$ (wt. %)	Fe_3C (wt. %)
500	1	94	6	-
500	4	21	9	70
500	8	50	5	45
400	1,5	94	6	-

4. Conclusion

Thus, the possibility of using low-energy electron beams for plasma cementation of stainless steel has been shown. The proposed method allows independently and within wide limits to change various processing conditions: temperature, pressure and composition of the gas mixture, the energy of the ion flux. At the same time, an important parameter that determines not only the speed, but even the possibility of forming a hardened layer, is the value of the flow of acetylene.

Acknowledgments
The authors thanks to A.I. Medvedev for X-ray phase analysis of samples and Yu.S. Surkov for the optical emission spectroscopy researches. The work was financially supported in part by the RFBR (grant No. 18-38-00561_mol_a).

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